THE ANALYST.

DECEMBER, 1892.

TO OUR READERS.

In consequence of the great pressure on the space hitherto available in The Analyst, and of the impossibility which has existed of reproducing many articles of interest and utility to Analysts, it has been decided to permanently enlarge the Journal with the commencement of the new volume in January next. From that date the price to annual subscribers, in advance, will be 10s. 6d. per annum, post free; the price for single numbers being one shilling. As there are very few back volumes of The Analyst in existence, the price of these will be from this date one guinea each.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

A Meeting was held at the Rooms of the Society of Arts, John Street, Adelphi, on Wednesday, the 2nd of November, the chair being occupied by the President (Mr. O. Hehner).

The minutes of the last meeting were read and confirmed, and papers read on the following subjects:—"Note on a sample of poor, but undoubtedly genuine, Milk," by Mr. C. W. Heaton; "An abnormal sample of Milk," by Dr. Bernard Dyer; "Composition of an abnormal sample of Milk," by Mr. Alfred Smetham; "Some abnormal Milks," by Mr. H. Droop Richmond; "An abnormal sample of Milk," by Mr. W. Foulkes Lowe.

The reading of these papers was followed by a long and animated discussion.

Mr. H. D. Richmond then exhibited and explained the working of

AMAGAT AND JEAN'S OLEOREFRACTOMETER.*

Dr. Muter having been asked (as the original introducer of theinstrument to British Analysts) to give his experience of its working, said:—

He had no doubt of the general utility of the instrument, and he considered it a profitable investment on the part of any public analyst in considerable practice. Like

* For full description and illustration, *see* The Analyst, vol. xv., page 87.

every other special appliance, it had its advantages and its drawbacks. Its use depended to a considerable extent on "personal equation," and on the exercise of care in the selection and preservation of the typical oil employed. Analysts using it should make their own standard observations, and not go blindly upon the published figures of other observers. So long as the instrument was only used for oils, the typical oil once placed in the outer casing of the prism would keep good for a long time, but when solid fats (such as butter) were worked with, the continued heating and cooling of the oil caused it to become cloudy, owing to the crystallization of the stearin, and then wrong results were obtained. Under such circumstances it was necessary to change the oil more frequently, and to make a test of the zero with fresh typical oil in the inner prism each day before commencing work. In dealing with butter it was undoubtedly better to heat the sample 20 degrees above the required temperature, place it in the prism, allow it to gradually cool down, and then to read the moment that the two thermometers marked the same degree. The vital point was that the oils inside and outside the prism must both be exactly at the same temperature. It was a very useful instrument for sorting butters, and so long as the present limit of "Reichert" continued to be a force, it would be found of no use to go on with any ordinary butter showing between 28 and 34 on the oleorefractometer. If over 34 or under 28 "Reichert" would be necessary, in the former case to prove a possible addition of cocoa-nut fat, and the latter for small percentages of ordinary fat. If the refraction was 20, or under, then the article must be at least a half-and-half margarine, and no "Reichert" was necessary to follow to confirm, as the sample could be definitely charged with being what is legally called margarine, and not butter. The practical use of the instrument was, therefore, to rapidly detect all cases of undoubted margarine, and also of definitely pure butter, but for any doubtful cases "Reichert" must follow and be considered side by side with the refraction.

This terminated the proceedings of the Society.

THE ACTION OF SOME ENZYMES ON MILK-SUGAR.

By H. Droop Richmond.

(*Read at the Meeting on October 5th*, 1892.)

These short notes formed part of an investigation of the methods for the complete analysis of milk and milk products. They were originally intended to see if rennet had any action on milk-sugar; it seemed very unlikely that this was the case, but being unable to find any experiments actually proving this, I decided to try for myself. I was also led to investigate this on account of Pappel and myself having isolated from gamoose milk a sugar, towfi'ose (tewfikose) by a method which avoided the use of rennet, and it appeared just possible that milk sugar might be formed from some compound existing in the milk by the action of rennet.

I assume that all sugars having the same rotary and reducing powers as milk-sugar are that compound, and that if the rotary and reducing powers do not alter, that the milk-sugar is unchanged. This assumption, I take it, will be granted by all.

The polariscope used was the Mitscherlich half-shadow polariscope described by Vieth (ANALYST, xi. 141); it is not a very delicate instrument, but I find that I can read to ·025°, by taking the average of several (say 10) readings. In the paper referred to Vieth states that the readings may be taken as giving grams of milk-sugar in 100 c.c. This, I find, is not quite, though nearly accurate. The tube used has a length of 198·2 millimetres, and the scale is divided into angular degrees; taking the specific rotary power $[\alpha]_D$ of milk-sugar to be 52·5° (*c.f.* Wiley, *Amer. Chem. J.*, vi. No. 5) the readings would require to be divided by 1·042 ($\frac{100 \times 100}{198{\cdot}2 \times 52{\cdot}5} = \frac{1}{1{\cdot}042}$). I have made many experiments to determine the actual factor, and find that the readings require to be divided by 1·045 to give grams per 100 c.c. These two factors agree well within the limit of reading.

I find, on the examination of the published statements as to the reducing power of milk-sugar, that some diversity of opinion occurs as to the method of obtaining accurate results. Thus, Soxhlet (*J. f. Prakt. Chem.* [2] xxi. 277, *et seq.*) finds that the same results are obtained whether the Fehling's solution be diluted or not; the conclusion of Rodewald and Tollens (*Ber.*, xi. 2076) does not quite accord with Soxhlet's. Muter (ANALYST, v. 35) indicates that, in order to ensure constant results, the sugar must be diluted till the solution contain less than ·1 gram in 100 c.c., and a very slight excess of Fehling's solution must be added. Jones (ANALYST, xiv. 81) employs O'Sullivan's method, in which a large excess of Fehling's solution, diluted with two measures of water, are employed, and brought to the temperature of the water-bath before the sugar is added, and the heating is continued for 14 minutes.

I have made some experiments on this subject, and find that O'Sullivan's method gives the most concordant results. I also find that, if the Fehling's solution is diluted with an equal bulk of water, I obtain the same results as when diluting with two volumes of water, and there is less solution to be filtered. I collect the precipitate of Cu_2O on a filter, and burn first over a bunsen and then in a muffle. Duplicates do not differ more than about 1 milligram. I find that the filter absorbs 2 milligrams from the Fehling's solution, and therefore deduct this. Jones gives the factor to convert copper oxide into milk-sugar (anhydrous) as 0·6024. I myself find 0·6025, and calculate from the mean of Rodewald and Tollens' results the factor 0·6022.

The method and factor given in the valuable paper of Jones are therefore to be relied upon.

My first experiments were made by taking milk and estimating therein the milk-sugar, and estimating the milk-sugar in the whey produced by rennet in the same milk, and calculating the proportion of the milk-sugar to 100 parts of water in each.

The mean of three experiments gave—

	Milk.	Whey.
Milk-sugar (polarised)	5·30	5·31
„ (gravimetric)	5·28	5·28

Rennet has therefore no action on milk-sugar, and milk-sugar exists in the milk as the same compound which exists in the whey.

I next made a solution of milk-sugar; 25 grams of finely-powdered milk-sugar, which had been very carefully purified by Dr. Vieth, were shaken for five minutes with 450 c.c. of cold water, and immediately filtered, and the solution mixed and polarized. The readings were taken at from 7 to 8 minutes after the addition of the sugar to the water; after a further ten minutes, the readings were again taken; and again at intervals of quarter-hour, half-hour, one hour, six hours, and twenty-fours. Taking the first reading to have been at an interval of five minutes from the mean time of solution, I constructed a time-curve for the fall in rotary power, and from this calculate that at the moment of solution the specific rotary power is 82·0 ± ·2. I also estimated the density of the solution at 15·5° after an interval of ten minutes, and after an interval of twenty-four hours. In each case it was 1·0193, showing that no change in the density had occurred concurrently with the change in the specific rotary power.

This solution contained 5·20 grams in 100 c.c. of milk-sugar determined by evaporation, 5·22 grams by the polariscope, and 5·18 grams from the weight of the copper oxide reduced, and 5·17 grams from density.

With this solution the following experiments were tried:—

Action of rennet.—To 50 c.c. of this solution 10 milligrams of rennet were added, and this was kept at 40° C for one hour. After cooling and filtering, it gave the following:—

Sugar (polarised)	5·22
„ (gravimetric)	5·23

This shows again that rennet has no action on milk-sugar.

Action of pepsin.—To 50 c.c. of this solution 5 c.c. 1% hydrochloric acid and ·03 grams of pepsin were added, and the solution heated for 3 hours at 40° C. After cooling, filtering, and correction for the small figures obtained in a blank experiment, the following figures were obtained:—

Sugar (polarised)	5·24
„ (gravimetric)	5·19

Pepsin is without action on milk-sugar.

Action of pancreatic extract.—To 50 c.c. of this solution were added 1 c.c. of pancreatic extract, and ·05 grams of sodium bicarbonate, and the whole digested at 55° C for

one hour. After cooling, filtering, and correction for the small figures obtained in a blank experiment, the following figures were obtained:—

Sugar (polarised)	5·22
„ (gravimetric)	5·17

Pancreatic extract has then no action on milk-sugar.

In the experiments quoted in no case was the enzyme destroyed by heating before estimating the sugar. It was found, in all cases, that while the reducing power was only slightly affected thus, the specific rotary power was diminished (in the case of pepsin digestions the acid being first neutralized). Experiments on milk showed also that the specific rotary power of the sugar is reduced by heating. The study of the changes of milk-sugar by heat is reserved for a future communication.

These experiments show that the milk-sugar in whey and in artificially digested milk can be accurately estimated, provided that they have not been heated.

The change of rotary power on heating milk-sugar (in solution) shows that some chemical change is taking place. This sheds some light on the fact that constant results cannot be obtained on continued drying of the total solids of milk unless the water is rapidly evaporated. I have noticed that if precautions are taken to get rid of the bulk of the water in a short time (*c.f.* ANALYST, xiv. 121) that the residue does not turn brown on continued heating. As in ordinary total solid estimation about one hour elapses before the residue is apparently dry, there is sufficient time allowed for considerable decomposition, which may (and probably does) render the results inaccurate. On this point I believe that my investigation of the changes of milk-sugar on heating will shed considerable light.

[NOTE.—With reference to the slight error involved by taking the reading of the Mitscherlich half-shadow polariscope to indicate grams of milk-sugar per 100 c.c., Dr. Vieth writes me that he has long been aware that it is not correct; but as the error introduced is small, he has preferred to call the actual readings milk-sugar on account of practical convenience. The mean of Dr. Vieth's readings on accurately prepared solutions of milk-sugar gives the factor 1·044.—H. D. R.]

THE ESTIMATION OF TOTAL SOLIDS IN MILK.

PRELIMINARY NOTE BY H. DROOP RICHMOND.

(*Read at the Meeting on October 5th*, 1892.)

THE proportion of total solids in milk is always estimated by evaporating the water, and weighing the residue; the most usual method is that adopted by the Society of Public Analysts, to evaporate 5 grams of milk in a basin to practical constancy.

A very superficial observer cannot but be struck by the fact that the residues are always more or less brown in colour, and the sole inference that can be drawn from this

fact is that some decomposition takes place. This was realized by Wanklyn more than 20 years ago, and to avoid this he recommended that the limit of drying should be three hours, in which time he assumed the water to be completely driven off, and no decomposition to have commenced; he assumed that by three hours drying the milk-sugar was not dehydrated. The results of Erdmann, Schmoeger, and Urech render this last assumption unlikely. It is evident from the application of simple physical laws that constant results cannot be obtained thus, unless either a large margin of time be allowed, or unless various conditions, such as the hygrometric state of the air and the rate of evaporation, can be kept constant; neither of these conditions obtain in practice, and therefore Wanklyn's method cannot be included among those to which consideration need be given.

Among other methods for the estimation of total solid may be mentioned those of Gerber and Radenhansen who curdle the milk previously to evaporation by the addition of either acetic acid or alcohol; they give the preference to alcohol on account of its greater rapidity and accuracy; within late years the use of acetic acid was resuscitated, and I believe claimed as original, by Johnstone.

Duclaux considers that sponge is the best medium on which to dry the residue; he employs a U-tube of peculiar shape immersed in boiling water, through which a current of air is aspirated. Gannter uses wood pulp in a basin. Adams' paper method is too well known to need any description. Babcock and Macfarlane have each described substantially the same method of drying the residue on asbestos contained in a glass tube. Muter and others dry on sand, and Muter has also used copper oxide for the purpose.

Storch uses pumice as a medium for evaporating milk.

I have proposed drying about 1 gram in a wide platinum basin, spreading well over the bottom during the evaporation.

It is noticed that if the milk is greatly spread out and the evaporation proceeds very quickly, that the residue remains white, showing that the same decomposition is not taking place.

I find that none of these methods are free from objection; Gerber and Radenhansen's methods do not give a white residue, and the residues lose weight almost indefinitely on drying continually. The same applies to the S. P. A. method, and for this reason it is directed to dry to practical constancy. It is at once evident that one experimenter's idea of practical constancy may not be the same as another's, and results may and do differ somewhat. The methods of Duclaux, Gannter, and Adams necessitate the previous drying of somewhat hygroscopic substances. The methods of Babcock, Macfarlane, and Muter involve the use of apparatus which is not so convenient as a basin, while in my own method so small a quantity must be used that errors of weighing

are greatly multiplied. The use of sand and pumice I find does not sufficiently effect the spreading of the milk over a large surface.

For these reasons I have tried the use of asbestos contained in a platinum dish; I place about 3 grams of asbestos (of the best quality) in a platinum basin, and ignite;* I weigh this, add 5 grams of milk, and dry on the water bath for about two hours, and then leave it in the air or water-oven at about 98° for twelve hours or more (usually over night); I find that I can obtain absolutely constant weight, a further drying for 24 hours, even at 105°, does not affect the weight as much as a milligram, and as some of the weighings are less and some more, I attribute any minute difference to errors of weighing; the residue serves excellently for the purpose of ash estimation.

I find that this method agrees very well in duplicate, and with my former method, and is about 0·1 per cent. higher than my own S. P. A. method determinations; I have used it on samples which were analysed in duplicate by two other chemists by the S. P. A. method, and it agrees almost exactly with the results of one, but is slightly lower than those of the other; my S. P. A. results are the lowest of the three, indicating that in the S. P. A. method there is a personal error to be considered. I do not wish to attack the S. P. A. method, as the differences are small and rarely exceed ¼ per cent., which is in practice a very good agreement, but am endeavouring to find a method which can be relied upon for scientific purposes; I do not yet claim that the method described above is accurate, as I am fully aware that agreement of duplicates, prevention of browning of the residue, and constant results do not prove accuracy, though the contrary holds. I am working to obtain proof that this method is accurate, and have brought this forward as a preliminary notice, in order that my fellow experimenters may also test it, and I shall be happy to exchange samples with any chemist who may communicate with me, with a view to test it, and to see if there is a personal error.

ON THE BABCOCK METHOD OF MILK ANALYSIS.

By Frank T. Shutt.

In a paper recently read before the Society of Public Analysts on the Determination of Fat in Milk, Mr. Stokes quotes *some* of my results obtained by the Babcock method, neglecting others, and entirely ignores my conclusions. My paper on this question has already appeared *in extenso* (*Chem. News*, lxiv., 4), so I could not expect you to publish it. I would, however, request space to place before your readers such extracts from it as will make clear my reason for believing this to be a most admirable dairy process.

Examination as to Reliability and Accuracy.

Thirty-two samples of milk have been examined in duplicate by (1) the Babcock test and (2) by gravimetric analysis, the results by the latter being taken, for the sake of comparison, as correct. These operations gave us 128 independent fat determinations.

* To ignite asbestos to constant weight, it is necessary to use a muffle, since by ignition over a bunsen or blowpipe the total combined water is not always lost.

As the results obtained throughout the whole series are uniformly close, it will suffice here to tabulate a few of them as examples :—

No.	Percentage of Fat by Babcock method.	Percentage of Fat by gravimetric analysis.
1	4·4	4·54½
	4·4	4·56
2	3·4	3·58
	3·5	3·56
3	3·6	3·72
	3·5	3·76
4	4·8	4·90
	4·8	4·91
5	5·8	6·04
	5·9	6·07
6	3·5	3·40
	3·3	3·35
7	3·4	3·60
	3·5	3·62
8	3·8	3·68
	3·7	3·68
9	3·8	3·87
	3·9	3·88
10	4·0	4·04
	4·0	4·03
11	3·3	3·32
	3·3	3·33
12	3·5	3·70
	3·7	3·69

An inspection of this table will reveal (1) the limits within which the amount of fat will vary when the Babcock test is made in duplicate, and (2) the approximation of such results to those given by carefully conducted chemical analyses.

Of the thirty-two samples tested in duplicate by the Babcock method, only two gave a difference between their duplicates amounting to three-tenths (·3) of one per cent. ; two varied in their duplicates two-tenths (·2) of one per cent. ; fourteen differed to the extent of one-tenth (·1) of one per cent., and thirteen gave results identically the same.

The greatest difference between fat determinations by the Babcock test and gravimetric analysis on the same milk was (·25) a quarter of one per cent. This occurs in three instances only. Where the results are not identical, the variation is usually between one-tenth and two-tenths of one per cent.

From these data therefore we may safely conclude that when the Babcock test is made according to the instruction given with the machine, *strictly reliable results are*

obtained, and that the percentage of fat so found, allowing for the greatest error possible under such circumstances, will be well within one-quarter of one per cent. (·25) of the amount of fat actually contained in the milk.

Since the publication of the above, I have made many trials towards applying the method to the testing of composite samples, and have met with gratifying results, as the following record, taken from many others of a like degree of accuracy, will show. In addition to the usual Babcock apparatus, a special pipette delivering 2·92 c.c. is all that is necessary—no preservative, poisonous or otherwise, being requisite to the accurate working of the test. To obtain the average percentage of fat in a cow's milk, or of milk supplied by the vendor, for a period extending over six days, successive measures of milk should be delivered from this pipette daily into an ordinary Babcock bottle. This composite sample is then treated with sulphuric acid in the usual manner. The percentage of fat so found is equivalent to the mathematical average of daily trials. The curdling of the milk in the bottles before the expiration of the period—which always occurs—apparently in no way vitiates the accuracy of the result.

Day.	Quantity.	Percentage of Fat.
Monday	17·6 c.c.	3·5
	17·6	3·6
Tuesday	17·6	3·3
	17·6	3·2
Wednesday	17·6	5·4
	17·6	5·4
Thursday	17·6	4·7
	17·6	4·7
Friday	17·6	3·4
	17·6	3·4
Saturday	17·6	4·6
	17·6	4·6
Average of above		4·15
Monday to Saturday inclusive, composite ...	2·92	4·2
	2·92	4·2

It is scarcely necessary to remark that this modification effects a great saving of time, while I think it is safe to infer from the above results that it entails no loss of accuracy.

The Babcock method, using composite samples, offers a cheap and reliable means of estimating fat in milk, and undoubtedly in the near future it will be widely used in all places where milk is valued and paid for according to its fat contents.

Chemical Laboratory of the Dominion of Canada Experimental Farms, Ottawa.

The use of Centrifugal Machines in Analytical and Microscopical Work. W. Thorner. (*Chem. Zeit.*, 1892, xvi., 1101–1104.)—The author has, on former occasions, shown the utility of a centrifugal apparatus for facilitating analytical and microscopical work (ANALYST, 1891, p. 210), and has now collected his experiences in a single article, to which additions based on subsequent experiments have been made. The instrument he uses is the Victoria Separator, by Watson, Laidlaw & Co., of Glasgow. A modification of the instrument has been introduced, consisting in an iron case with a removable cover surrounding the centrifugal plate, bearing the tubes containing the substances under treatment, which is designed to prevent accident should breakage occur. No such accident has happened in the author's experience; but the case is useful as lessening the resistance to rotation on account of the air inside the box being rotated with the tubes, and therefore stationary relative to them. The centrifugal plate, which is a stout, well-tinned brass casting, is made in two sizes, 160 and 240 mm. in diameter, and is fitted with metal tubes or cases, with an internal diameter of 19 and 36 mm., for the reception of the glass tubes in which the operations are conducted. The larger plate is arranged to take 8 small cases, 16 mm. in diameter, for fat determinations in milk, while the smaller plate is provided with two of the larger and two of the smaller cases, so as to adapt it for more general purposes. Special larger cases can also be used, with a capacity of as much as 400 c.c.; but when using vessels of this size, care must be taken to balance the machine with another cylinder of similar weight, a precaution of general applicability. The cylindrical metal cases for the reception of the glass tubes are made of well-tinned sheet brass or copper, and are provided with a cotton pad, to avoid risk of breaking the tubes dropped into them. Tubes, of which the lower part is narrowed and graduated, are supported in a wooden block, similarly shaped, and slipped into the metal case.

The use of these various apparatus is shown by the following examples:—

(1.) *Determination of fat in milk and milk products.*—10 c.c. of the well-mixed sample are placed in a tube with a narrow graduated neck, 1·5 c.c. of alcoholic potash containing 160 grammes of KOH per litre, or 1 c.c. of an aqueous solution of 500 grammes KOH per litre, added and mixed therewith by gentle shaking. The tube has a wide collar above the narrow neck, and it is hung by means of this in a boiling water-bath, the screw-clip on the rubber tube carried by the cork in its mouth, being closed after 10-15 seconds, when the alcoholic liquid (if that has been used) begins to boil. The tube is removed from the bath after 2-3 minutes, again shaken gently, and glacial acetic acid run in by means of a tap funnel until the liquid is above the zero mark on the narrow neck. Further shaking, and the addition of more acid may be necessary to complete the solution of the casein. The rubber cork is then replaced, and the tube again hung in the water-bath, from which it is removed after some minutes, the screw-clip opened, the cork taken out, and the tube rotated in the separator at 2,000 or 3,000 revolutions per minute,

according as the larger or smaller plate is being used. The tube is then returned to the bath, and brought to a temperature of 100° C. before reading off the fatty acids. The results agree with those obtained by gravimetric methods. Eight determinations of fat can be made in twenty minutes.

(2.) *Determination of cream.*—This can be done by 10-15 minutes' rotation of 20 c.c. of milk. The end is more easily attained by diluting the sample with an equal volume of water, and doubling the observed reading.

(3.) *Determination of the contents of water in butter and other fats.*—The lower part of the centrifugal tube is contracted and graduated. 10 c.c. of the fat are used, and the results up to a content of 30 per cent. by volume are exact to 0·2 per cent. The butter is measured by the little apparatus described in a previous abstract. (THE ANALYST, 1891, p. 210.)

(4.) *Determination of fat (or more strictly fatty acids) in butter and other fats.*—A tube with a narrow graduated neck, like that used for milk, but without the stopper and screw-clip, is used. The same apparatus serves for the determination of fatty acids in soap, and the content of mineral oil in mixed lubricants.

(5.) *Testing flour and meal.*—The apparatus for the separation of various flours and meals has already been described (THE ANALYST, p. 210, 1891). The microscopic examination of meals and starches is facilitated as follows : 5-10 gms. of the meal are stirred with a little cold water, poured into 500 c.c. of hot water, and heated for half hour on the water-bath. About 100 c.c. of the well-mixed contents of the flask are treated with 1 c.c. of 50 per cent. KOH solution, and another similar quantity with 5 c.c. of glacial acetic acid, and again heated on the water-bath. The liquid is then rotated in a plain cylindrical tube for five minutes, and the resulting residue at the bottom of the tube examined microscopically.

(6.) *Examination of tuberculous sputum.*—The sputum is mixed in a plain cylindrical tube, after the addition of at least an equal volume of hot water or dilute solution of borax, well shaken, heated in the water bath for ten minutes and rotated briskly for 5—10 minutes. The residue at the bottom of the tube is then examined according to known methods. Very viscous samples are best treated by the addition of a few drops of 50 per cent. KOH solution before heating and rotating. The residue can be washed, if thought necessary, by subsequent rotating with water.

(7.) *Examination of milk for tubercular bacilli.*—20 c.c. of the sample are mixed with 1 c.c. of 50 per cent. KOH and rotated in a plain cylindrical tube, heated for two minutes, 20 c.c. of glacial acetic acid added, the whole well mixed, heated again for three minutes until the casein is dissolved, and rotated for ten minutes at 3,000 revolutions per minute. The residue is washed by rotation with 40 c.c. of hot water for ten minutes, and then examined microscopically, as usual.

(8.) *Examination of urine.*—50 c.c. of the sample are placed in a tube with the lower part contracted and finely divided, so that its total capacity is 0·5 c.c. and 0·005 c.c. can be read, and rotated briskly for five minutes. Should the urine be only slightly turbid a larger quantity, *e.g.*, 100 to 400 c.c., must be used in a larger tube, the residue from which is then treated in the smaller tube with the fine divisions. The residue is examined microscopically. The author has succeeded in detecting tubercular bacilli in urine, with and without staining. The method would probably be available for the determination of albumen, but experiments on this point are not yet complete.

(9.) *Determination and examination of turbidities in wine, beer, etc.*—The methods used for urine are applicable here.

(10.) *Examination of water.*—100 c.c. of the water to be examined are rotated for ten minutes in order to induce the ready settlement of any bacilli, etc., that may be present; a little fine sterilised elutriated clay or talc is added, about 0·1 c.c. being shaken up with the water before rotation. Waters naturally slightly turbid with clay or ferric hydrate, of course, need no such addition. The residue is washed by rotating it in a narrow tube, the bottom of which is contracted like that used for urine, with 30—50 c.c. of water for ten minutes. The finest residue is used for the preparation of plate cultivations on alkaline or slightly acid gelatin in the usual way.

(11.) *Volumetric determination of precipitates in analysis.*—When the quantity of liquid is small, *e.g.*, under 50 c.c., the precipitation can be conducted in the tube with the contracted lower part, already mentioned, and the volume of the precipitate read off after rotation. In the case of larger quantities a larger tube is first used in which the ordinary operations usually carried out in a beaker or flask, including even heating (with caution) over a naked flame, are performed, and the precipitate transferred after rotation to the smaller tube, and treated as before. In order to obtain accurate results a standard time and rate of rotation must be adopted.

The apparatus described above can be obtained from Dierks & Möllmann, of Osnabrück. B. B.

Influence of the Sulphur in Coal Gas on Determinations of Sulphur by Fusion. Van Leeuwen. (*Rec. Trav. Chim. Pays-Bas*, 1892, xi. 103, through *Chem. Zeit.*)—The author's experiments were carried out on spent gas-works purifier, of which 0·5–0·6 was treated with 35 grms. (!) of a mixture of 1 part of potassium chlorate and 6 parts of sodium carbonate, in a platinum dish either covered with a porcelain lid, or with another inverted platinum dish, for 2½ hours over a bunsen burner. The percentages of sulphur found in three determinations were 49·34 per cent., 48·55 per cent., 47·98 per cent., giving a mean of 48·63 per cent. Two blank experiments showed that a quantity of sulphur was absorbed from the gas amounting to 2·4 per cent. and 3·3 per cent. reckoned on a 0·5 grm. of the substance in which sulphur was to be determined.

As the coal gas used did not blacken lead paper, it appeared that the sulphur was present as carbon disulphide, or some analogous compound, and the author repeated the experiment, using in one case the unpurified gas, and in the other gas that had been passed over pumice saturated with an alcoholic solution of caustic potash. The first gave an amount of sulphur corresponding to 4 per cent. of sulphur reckoned on 0·5 grm. of the substance to be analysed, while the second only yielded a quantity equivalent to 0·1 per cent. Three determinations of sulphur in the spent purifier, of which the figures obtained with ordinary coal gas are given above, gave 46·97 per cent., 46·26 per cent., and 46·29 per cent., that is a mean value of 46·50 per cent., against the former result of 48·63 per cent. The author recommends for such purposes as those described the new Barthel's burner, fed with benzine free from sulphur, or with alcohol. B. B.

[Note by Abstractor.—The errors here detailed are real and serious, but they can be most readily and certainly avoided by the use of the gas muffle.]

The Gravimetric Determination of Sulphur. M. Ripper. (*Zeits. anorg. Chem.*, 1892, ii. 36, through *Chem. Zeit.*)—The reduction of barium sulphate to sulphide, which the author considers to take place when the former substance is incinerated according to the modern method of placing the precipitate in a crucible together with the filter paper, both in a moist state, and slowly drying and burning, can be conveniently remedied by the addition of a few drops of bromine water, until it ceases to be decolorised, before the purification of the barium sulphate with hydrochloric acid. This treatment with bromine water is, of course, repeated after the barium sulphate has been purified with hydrochloric acid and again incinerated. B. B.

A New Process for the Separation of Iron. J. W. Rothe. (*Mitt. könig. tech. Versuchsanstalten*, 1892. x. 132, through *Chem. Zeit.*)—The process depends on the observation that ferric salts in hydrochloric solution of suitable concentration, yield the whole of their iron as ferric chloride when shaken out with ether. Ferrous salts are not dissolved out by ether under like conditions. Manganous salts, chromic salts, and those of nickel and aluminium, give no soluble compounds when thus treated. Cobaltous and cupric salts are soluble to nearly the same extent in ether, the exact amount varying with the quantity present, but it is in any case very small. What is extracted can be got rid of by shaking out the ethereal solution of ferric chloride with hydrochloric acid of specific gravity 1·04, and the small quantity of iron thus extracted can be recovered by renewed treatment with ether.

In the case of iron and steel, the method is worked as follows:—Five grms. of the sample are dissolved in hydrochloric acid of specific gravity 1·124 at 19°C, heated on the water bath, and then taken to dryness, finishing in an air bath at 120°C. The residue is

treated with 20 c.c. of the same hydrochloric acid, heated to boiling, diluted with three times its volume of water, and filtered from silica. The filtrate is evaporated to a small volume until hydrochloric acid begins to escape, 10 c.c. of concentrated hydrochloric acid added, and then oxidised with 2–2·5 c.c. of nitric acid of specific gravity 1·4, added drop by drop to the boiling liquid. The product is then evaporated on the water bath to a syrupy consistency, or until a basic chloride begins to separate. The resulting solution, which should measure about 10 c.c. is transferred to a specially constructed separator, and 55–60 c.c. of hydrochloric acid of the strength mentioned above, added, and the whole shaken out twice with ether. Should copper or cobalt be present, its removal must be provided for in the manner already described. When the separation of iron and aluminium is effected by this process, particular care must be taken that the solution in the separator is quite clear and free from any basic salts of the latter metal. The author gives analytical figures showing the reliability of the process, and states that it has been used in more than 100 analyses of iron, steel, and ferromanganese, with successful results. B. B.

New Colour Standard for Natural Waters. A. Hazen. (*Amer. Chem. J.* xiv. 300-310.)

Leeds suggested the adoption of nesslerised ammonia as a standard for comparison with the colour of natural waters, degrees of colouration being expressed by the number of c.c. of dilute ammonia solution (0·01 mgm. NH_3 per c.c.) which, when nesslerised in 50 c.c. of distilled water, match the colour of the natural water. An insurmountable objection to the method is the variable character of the colour obtained by nesslerising ammonia; both temperature and quality of nessler reagent influence the colouration.

Crookes, Odling and Tidy use an empty tube of the same length as that containing the water to be compared; this has two hollow wedges behind it, the one filled with a 1 per cent. copper sulphate solution and the other with a mixture of ferric chloride (0·7 grm. per litre) and cobalt chloride (0·3 gram. per litre) solutions with "a very slight excess of hydrochloric acid." These wedges are pushed over the empty tube until the colour, on looking down it, appears identical with that of the water. In this case degree of colouration is expressed as equivalent to so many mm. of blue, and so many mm. of brown solution. The colours of these solutions are, however, variable both with temperature and quantity of hydrochloric acid present.

The author proposes a platinum colour standard, the colour to be expressed as "the amount of platinum, in parts per 10,000, which, in acid solution, with so much cobalt as will match the hue, produces an equal colour in distilled water." The standards are made thus:—1·246 grms. of potassium platinic chloride (0·5 grm. Pt.) and 1 grm. of crystallised cobalt chloride (0·25 grm. Co.) are dissolved in water, 100 c.c. of strong hydrochloric acid are added, and the whole is made up to 1000 c.c. This stock solution will

remain unchanged for a long time, even when exposed to light. For comparison with waters, 1, 2, 3, etc., c.c. of the stock solution are diluted to 50 c.c. in Nessler tubes. These correspond to 0·1, 0·2, 0·3, etc., degrees of colour standard. These also keep for months, in absence of dust. If the hue do not match that of the water, more or less cobalt may be added, the platinum being kept constant.

Direct comparison in 200 mm. Nessler tubes is generally sufficiently accurate. But when the colour is of great importance, the author uses 600 mm. brass tubes (washed internally with lead acetate solution, to destroy reflection) with plate-glass ends; and instead of mixing the stock solution with the distilled water he places behind the tube containing the latter, flat jars of solution corresponding to 0·01, 0·02, 0·03, etc., degrees of colour. The solution in the jars must be as many times stronger than that which would have been used had the tube been filled with it, as the tube is longer than the thickness of the jar.

For waters more than very slightly turbid and of more than 0·3 degrees of colour, the 600 mm. tubes are not good. For those of more than 0·9° the 200 mm. tubes are not good. Between this and 1·8° 100 m.m. tubes may be used.

Some remarks on the variation of the absolute colour produced by Nessler's reagent with the same quantity of ammonia under varying conditions,—which, however, do not occur in the usual determination of ammonia,—and a comparison of the Nessler standard with the platinum standard for the colour of a water, conclude the paper.

A. G. B.

Estimation of Sulphur in Pyrites Cinders. G. Lunge. (*Zeits. angew. Chem.*, 1892, 447; through *Chem. Zeit.*)—The author conducts Watson's method as follows:—3·2 grams of cinders are mixed with 2 grams of sodium bicarbonate of known titre, and heated in a nickel crucible (20 grams capacity) for 10—15 minutes, so that the point of the flame shall only play upon the bottom of the crucible, the bicarbonate being thus decomposed. The heating is continued for 15 minutes over a stronger flame, so that the whole mass becomes red hot. Fusion is to be avoided, and the crucible must be kept covered. The cooled mass is turned into a porcelain dish; if the temperature has been high enough it will be black and porous, otherwise it will be glassy and difficult to dissolve. After digestion with hot water, a strong solution of sodium chloride, carefully made neutral to methyl orange by hydrochloric acid, is added to the solution in order to obtain a clear filtrate; for the same purpose the filter paper should be first washed with the salt solution. The liquid having been decanted on to the filter, and returned if it come through of a yellowish green colour, the residue is digested with more of the salt solution, and finally washed with a dilute salt solution. The filtrate and washings are titrated with pentanormal hydrochloric acid. The sulphur is calculated from the amount of bicarbonate which has been converted into sulphate, as determined by the decrease in alkalinity.

A. G. B.

Estimation of Acetone. H. Strache. (*Wien Monatshefte für Chemie* 1892, xiii., 299-315.)—The chemistry of this process may be summed up in the statement that the hydrazones, which phenylhydrazine forms with aldehydes and ketones, are incapable of oxidation, with liberation of nitrogen, by Fehling's solution, whereas phenylhydrazine itself is quantitatively so oxidised at the boiling point of the solution, with liberation of all its nitrogen. The readiest method, therefore, for determining the amount of hydrazone produced by the reaction between an aldehyde or a ketone, and excess of phenylhydrazine, is to boil the mixture with Fehling's solution and measure the evolved nitrogen.

A weighed quantity (probable excess) of phenylhydrazine hydrochloride is dissolved in warm water together with $1\frac{1}{2}$ times its weight of sodium acetate. A measured quantity of the liquid containing the acetone is added to the solution, and the whole is heated on the water-bath for 15 minutes. The volume is now made up to 100 c.c., and 50 c.c. are transferred to the separating funnel of the apparatus described in the following paragraph, the stem of the funnel having been previously filled with water.

A convenient quantity (200 c.c.) of Fehling's solution is placed in a flask ($\frac{3}{4}$—1 litre) which is provided with a three-holed cork. One of the holes carries a tube which passes to the bottom of the flask and is connected with another flask for supplying steam. The second hole is for the separating funnel; the end of this reaches to the bottom of the flask and is drawn out and curved upwards to prevent gas from escaping up the stem. Through the third hole passes the tube which is to deliver the nitrogen into the measuring tube.

The Fehling's solution is heated to the boiling point and a rapid current of steam is passed through the apparatus, until the air has been expelled as completely as possible. The phenylhydrazine mixture is then allowed to flow from the funnel into the flask very slowly at first, in order to avoid cooling the liquid in the flask and creating a back-suck. The nitrogen is very rapidly evolved and swept on by the current of steam, the whole operation only occupying two or three minutes. Inasmuch as benzene is a product of this reaction, the nitrogen will contain the vapour of that compound; allowance must be made for this in measuring the gas, which is best saturated with the vapour by allowing a few drops of benzene to rise up the water in the measuring tube. The following are the corrections to be made for the tension of benzene + water at various temperatures :—

At 15°	72·7 mm.	At 21°	98·8 mm.
„ 16°	76·8 „	„ 22°	103·9 „
„ 17°	80·9 „	„ 23°	109·1 „
„ 18°	85·2 „	„ 24°	114·3 „
„ 19°	89·3 „	„ 25°	119·7 „
„ 20°	93·7 „		

The difficulty of completely expelling the air from the flask tends to make the volume of the gas measured too large. Thus, the author found, as a mean of 8 experiments, that 1 grm. of phenylhydrazine hydrochloride gave 159·7 c.c. of nitrogen instead of 154·6 c.c. This error is, however, nearly constant for the same flask and is best avoided by calibrating the apparatus. This can be done by performing a blank experiment with 1 grm. of phenylhydrazine hydrochloride and substituting the volume of gas obtained, for 160 in the following formula, in which A is the weight of the acetone in grms., g the weight of the phenylhydrazine hydrochloride taken, Vo the corrected volume of the nitrogen evolved, and 0·002595 the acetone equivalent to 1 c.c. of nitrogen :—

$$A = [160g - 2\ Vo]\ 0{\cdot}002595$$

The acetone from a very dilute solution may be determined by boiling the liquid in a flask provided with a small absorption tube containing the phenylhydrazine solution. A current of air will hasten the distillation of the acetone into the phenylhydrazine.

The author hopes to apply this method to the determination of aldehyde, and possibly of alcohol by first converting it into aldehyde. A. G. B.

A New Method for the Determination of Carbon in Organic Substances. **K. Okada.** (*Arch. Hyg.* xiv. 364, through *Chem. Zeit.*)—The author has found that when organic substances are heated with sulphuric acid, as in the Kjeldahl process, only carbon dioxide is formed, no other volatile compounds containing carbon being evolved. This circumstance makes it possible to determine carbon by the method in question, and in the case of substances containing nitrogen, to estimate that element at the same time and by the same operation. The flask in which the operation is conducted, is provided with a glass tube ground-in, connected with a wash bottle, this in its turn is coupled with a second washing flask, and finally with a Pettenkofer's tube. The first wash bottle contains water, and the second permanganate, and the absorption tube is filled with baryta water to receive the carbon dioxide. At the end of the operation the apparatus is connected with a water pump, and air free from carbon dioxide drawn through it. The usual Kjeldahl mixture, of ordinary and fuming sulphuric acid, is used with the addition of a little mercury. The analytical results are concordant, and the process appears applicable to a number of substances. The author is engaged in devising a method of determining oxygen on the same lines. B. B.

The Examination of Commercial Yolk of Egg. **M. F. Jean.** (*Monit. Scient.* 1892, vi. 561, through *Chem. Zeit.*)—The author has confined his examination of fresh and salted yolk of egg to the determination of the water, the dry extract, the vitellin, the extractives soluble in water and the ash. The water is determined by adding a few drops of acetic acid to 10 grms. of the sample, drying first at 50°–60°C and then at 110°C, until the weight is constant. In order to determine the fat and the vitellin,

the dried residue is extracted with petroleum ether in a Soxhlet apparatus, the ether-extract evaporated to dryness, and the residue dried at 110°–115°C, and extracted with water. This aqueous extract is evaporated on the water bath and afterwards heated to 110°C. The residue thus left consists of water-soluble extractives which are taken as soluble vitellin. The residue insoluble in water is the fat. The ash is determined by incinerating 10 grms. of the sample in a platinum dish (at first at a low temperature), until the residue is white. When the water, fat, ash and water-soluble extractives are known, the difference is put down as insoluble vitellin, and this added to the soluble vitellin already estimated gives the total vitellin. The salts found in the ash are sodium chloride, sulphate, borate, and nitrate. Of these it generally suffices to determine the first, which is best done on the aqueous extract of a fresh quantity of the yolk of egg, dried at 100°C, rather than on the total ash. An average sample of fresh yolk of egg, analysed in the manner given above, contained water 52·6, ash, 1·4, fat 28, and vitellin 18 parts per cent. respectively. B. B.

The Rising of Cream in Watered Milks. Allard. (*Staz. Sper. Ag. Ital.*, xxii., 420, *from La Laiterie.*)—The author made comparative experiments as to the time taken by the cream in rising in a sample of undiluted milk, and the same milk diluted with 10, 20, 40, 50, 60 and 75 per cent. of water.

In genuine milk a distinct stratum was observed after 90 minutes (2·5 per cent.), while in that diluted with 50 per cent. of water 45 minutes only was requisite. The total quantity of cream (14·1 per cent.) was thrown up by genuine milk after 27 hours, by that diluted with 10 per cent. of water (13·3 per cent.) in 23 hours, 20 per cent. (12·5 per cent.) in 17 hours, and 40 per cent. or more in 2 or 3 hours.

The author remarks that if a milk throws up its cream in 3 hours the conclusion may be drawn that the pump has passed that way. H. D. R.

[Note by Abstractor.—Vieth has shown (Analyst, viii., 2), that some samples of genuine milk throw up their cream very rapidly; it is well known that no reliance can be placed on cream-tests on milk that has been refrigerated, and has had much shaking in transit, and this test is thus deprived of its value. As it will only give indications of extensive watering (40 per cent.), the lactometer would be much simpler and more reliable.] H. D. R.

Citric Acid. T. Pusch. (*Pharm. Zeit.*, 1892, xxxvii., 448; through *Chem. Zeit.*) —The author claims that citric acid may be stated to be free from lead when 50 c.c. of a 10 per cent. solution of it, contained in a beaker placed on a white surface, shows no darkening with sulphuretted hydrogen. Citric acid, absolutely free from lead, has been made in Germany for several years.

A. G. B.

Estimation of Lime Salts in Syrup and Sugar Products. J. Wolff. (*Oestr. ungar. Zuck. Ind.* xxi. 96—104, through *Chem. Centr.*)—The author effects his object by means of a soap solution. This is prepared from pure olive oil (150 grms.) by saponification with finely-ground litharge (100 grms.) and a solution of lead acetate (5 grms.) in water (40 c.c.) at 100°. The lead soap is washed, and after being softened on the water bath, 150 grms. of it are decomposed by trituration, with 40 grms. of potassium carbonate. The potash soap is extracted with alcohol, and the alcohol distilled off; 40 grammes of the residue are dissolved in a litre of alcohol of 56 Tralles (*sic*). This solution is then brought to such strength that 1 c.c. corresponds to 0·0005 grm. of CaO. The titrations necessary for this are effected thus:—100 c.c. of standard calcium chloride solution (0·15 grm. per litre), 25 c.c. of pure syrup, some drops of strong ammonia and water to make 150 c.c., are put into an Erlenmeyer flask, into which the soap solution is run until the addition of a drop produces a bright vesicular lather, about 1 c.m. thick, and permanent for at least 10 minutes. The operation must be repeated without the presence of calcium chloride, and the difference between the results held to be the number of c.c. equivalent to 100 c c. of the calcium chloride solution.

The estimation of lime salts in sugars is carried out in the same way, only care must be taken to obtain a genuine lather. To this end the presence of ammonia is desirable, and the quantity of soap solution necessary should not exceed 20 c.c.; the qualifications required of the lather, detailed above, must be insisted upon, and when it has disappeared it should be easily re-formed by another shaking. A. G. B.

Effect of "Centrifuging" on Bacteria in Suspension. (*Arbeit a. d. Kaiser. Gesundheits-Amte*, vii. (1891) through *R. M. S. Jl.*, 1892 p. 432.)—After having ascertained by experiments with anthrax that an hour's "centrifuging" at the rate of 2,000 to 4,000 turns per minute was not detrimental to the vitality or the virulence of these organisms, Herr Scheurlen turned his attention to the behaviour of bacterial pure cultivations in suspension.

The results of centrifuging were found to differ according as the bacteria were mobile or immobile, the latter being thrown out and forming a sediment, while some of the former, *e.g.*, Cholera Vibrio and Proteus Mirabilis, remained suspended.

The author then examined the behaviour of the bacteria of milk when similarly treated. After centrifuging, the milk serum, when tested by means of plate-cultivations, showed, as was to be expected, a large number of colonies; while the number in the cream was also very great, and might even exceed that of the serum. The author infers from these experiments that milk cannot be freed from its bacteria by centrifuging, for out of 2,050 million germs in the whole volume of milk, only 18 million were removed in the serum. About three-fourths of the number are transferred by the centrifuging to the cream, the remainder being in the butter-milk.

Most pathogenic microbes, such as anthrax, typhoid, and cholera, cling to the cream like the milk bacteria, but tubercle bacilli were for the most part separated out, only a few remaining in the milk and cream. F. H. P. C.

REVIEW.

COMMERCIAL ORGANIC ANALYSIS. By ALFRED H. ALLEN. Second Edition, Volume III., Part II. (London, J. and A. Churchill, 18*s*.)

We welcome the appearance of this further instalment of the Second Edition of Mr. Allen's great work. The matter has grown under Mr. Allen's hands to such an extent that Vol. III. will, when completed, consist of three separate volumes, of which the one under review comprises 570 pages, and treats of the Amines, Hydrazines, Tar Bases, and Vegetable Alkaloids, the last section occupying almost four-fifths of the volume. The chapters on the characters, reactions, and isolation of alkaloids convey in a most lucid manner an immense amount of practical information. The colour-reactions, in particular, are treated in an admirable manner, and more sound analytical information is given by the author in the pages devoted to this subject than in any work on toxicology with which we are acquainted. Mr. Allen enumerates in a concise way the various alkaloids which with dehydrating, oxidising, and other reagents give characteristic colours, and thus enables the analyst almost at a glance to identify an alkaloid under examination. The special chapters on aconite, opium, and cinchona alkaloids bear witness to Mr. Allen's thoroughness, and present exhaustive reviews of the most recent researches on these difficult and intricate subjects. The practical aspects are always kept fully in view, and the analyst will find all he may require in reference to morphiometry, the assay of barks, and the separation of cinchona bases. Of special interest to the public analyst is Mr. Allen's treatment of caffeine and its allies, under which head the composition and analysis of tea, coffee, and cocoa are considered in detail, and in a manner vastly more accurate and scientific than has been the case in any manual specially devoted to food analysis. The author's special knowledge of these subjects renders these chapters of the greatest value. Modern processes of precision will here be found recorded, and in many cases critically discussed. Mr. Allen's work appears to us the type of what a chemical treatise ought to be. Science and practice are blended on every page. We are fully familiar with purely chemical works to which the analyst often turns, and vainly so, for help and information when he has to solve a problem new to him, only to find that they too often avoid giving useful data to the practical worker. We are equally familiar with the practical works on analysis, especially of food, which are too often devoid of good, sound science. In Mr. Allen's volume, modern science and accurate description of the properties, chemical and physical, of the substances with which he has to deal are combined with concise descriptions of analytical processes, and with such information to the analyst that the work will be found a real and trustworthy laboratory friend. It is rare indeed that an author combines the scientific with the practical side as does the author of "Commercial Organic Analysis." The earlier edition of this work has been recognised to be of the utmost value to the practical chemist, and this second edition, which is in reality a new work, will be found indispensable to every analyst who desires to keep up with the rapid progress of organic analysis. We look forward with interest, and something like impatience, to the one volume now remaining to be issued.

O. H.